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13. ABSTRACT (Maximum 200 words) This project involved the procurement and installation of enhancements to Michigan Technological University's Molecular Beam Epitaxy (MBE) facility. MBE is a thin film deposition technique that permits precise control of multiple element fluxes to a surface, permitting alloy layers as thin as one monolayer to be prepared under carefully controlled conditions of vacuum and temperature. Unlike most other thin film preparation techniques, the deposition conditions for MBE can be far from thermodynamic equilibrium, allowing surface processes to govern transport and reaction kinetics either during or subsequent to the deposition.				
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Final Technical Report

Project Title: Instrumentation for Real-time Information Extraction from RHEED and Correlation Using Scanning Probe Microscopy: Applications to Si-Nano-electronics

Project Number: MTU #960101/ E20597

Sponsor: U.S. DOD/ARO

Principal Investigator: M. Krishnamurthy (deceased)

Co-Investigators: D. J. Swenson¹, T. J. Schulz

Introduction and Background:

This project involved the procurement and installation of enhancements to Michigan Technological University's Molecular Beam Epitaxy (MBE) facility. MBE is a thin film deposition technique that permits precise control of multiple element fluxes to a surface, permitting alloy layers as thin as one monolayer to be prepared under carefully controlled conditions of vacuum and temperature. Unlike most other thin film preparation techniques, the deposition conditions for MBE can be far from thermodynamic equilibrium, allowing surface processes to govern transport and reaction kinetics either during or subsequent to the deposition.

The MBE facility at Michigan Tech is used primarily for deposition of very thin films (primarily silicon and germanium alloy films) on mono-crystalline silicon and germanium substrates. It is the central materials processing facility used in research studying the processing conditions and atomic mechanisms associated with the formation of self-assembled nanostructures in these systems. These nanostructures include quantum dots, quantum wires, and quantum wells, all of which exhibit unique electronic and optical properties, not generally attainable through processing of comparable bulk alloys. Development of processing techniques for nanostructures, and the underlying knowledge of mechanisms by

¹ Technical questions regarding this report should be directed to Dr. D. J. Swenson, Department of Materials Science and Engineering, Michigan Technological University, Houghton, MI 49931

which they are formed, are essential if low cost integrated opto-electronic devices are to become a commercial reality.

Project Accomplishments:

The objectives of the enhancements were to permit increased rates of silicon deposition during processing, allow improved real-time characterization of the alloy films during the deposition process, and permit characterization of the films and self assembled nanostructures after they are processed by MBE. The three system components² procured and installed under this project were:

1. A high energy material source for the MBE system to permit increased deposition rates. This material source has been used to obtain a wide range of silicon deposition rates during growth of silicon alloy thin films on monocrystalline Si and Ge substrates.
2. Software and computers to upgrade the Reflection High Energy Electron Diffraction (RHEED) system used for real-time characterization of thin films as they are grown by MBE. RHEED creates a diffraction pattern of the top few atomic layers of a surface, which may be converted into a picture of the atomic arrangement of those layers using appropriate software. Any changes that occur in the arrangement of the surface atoms during MBE growth, including the generation of self-assembled nanostructures, may be observed by monitoring corresponding changes in the RHEED pattern. While its real-time characterization capabilities are valuable, RHEED cannot be utilized to image a specific feature on a growing surface; rather, it only provides an average picture of the atomic arrangement of the entire surface. Additional, ex situ characterization techniques are required to study the structural details of MBE-grown thin films and nanostructures.
3. An atomic force microscope (AFM) plus associated software and computers for characterization of as deposited films and self-assembled nanostructures. AFM is a scanning probe technique that allows the surface topography of as deposited films as well as nanostructures to be characterized on a scale approaching atomic dimensions. It provides much more detailed information about the films and nanostructures, but cannot be used during processing.

² A detailed list of items purchased using project funds, and their cost are provided in Appendix 1.

These enhancements to Michigan Tech's facilities for study of self assembled silicon based nanostructures have contributed directly to several significant developments in the field. These are described in several publications and two Ph.D. dissertations listed in Appendix 2. Mr. Jie Zhang, a Ph.D. candidate in Materials Science and Engineering at Michigan Tech received the Materials Research Society's Graduate Student Silver Award at its 1999 Fall Meeting for work that made extensive use of the MBE and related characterization facilities. Mr. Zhang's presentation was entitled "Manipulation of Self-Assembled Nanostructures of Group IV Elements by Molecular Beam Epitaxy".

Perhaps most noteworthy of the developments using this facility was the discovery of a process for making one dimensional quantum structures (quantum wires) on germanium substrates. This development, described in two refereed publications, one conference proceeding, and one Ph.D. dissertation, is listed in Appendix 2. It was also reported in the popular scientific and trade literature such as *Science* magazine³, *Discover* magazine⁴, *Popular Mechanics*⁵, *Advanced Materials and Processes*⁶, and *New Electronics*⁷.

Other Accomplishments

Although the enhancements to the MBE facility at Michigan Tech were intended primarily to facilitate research on nanostructured thin films as described above, the AFM has had the salutary effect of facilitating a wide range of research activities beyond those directed to thin films and electronic materials. Dr. J. W. Drelich has led these efforts, which have focussed on the use of the AFM to measure interaction forces between solids, and capillarity forces in liquid solid systems. Dr. Drelich has purchased a separate AFM head that can be operated using the computer and electronics purchased under this project. A number of graduate and undergraduate projects have been supported by this facility, and a list of publications based on that research is given in Appendix 3. These studies include topics as widely varied as de-inking of recycled paper, oil removal from contaminated soil, and measurement of adhesion forces for pharmaceutical particles on polymeric surfaces.

³ *Science*, January 1998, vol. 279, p. 991.

⁴ *Discover*, June 1998, p. 16.

⁵ *Popular Mechanics*, October 1998, p.24.

⁶ *Advanced Materials and Processes*, June 1998, p. 4.

⁷ *New Electronics*, October 1998, pp. 16-18 (also see cover).

Appendix 1 Equipment Purchased

Item Purchased	Vendor(s)	PO Number	Cost
High Energy Material Source for Molecular Beam Epitaxy	Thermionics Laboratory, Inc.	PO-032282	6,111.20
Reflection High Energy Electron Diffraction Upgrade	K-Space Associates, Inc.	PO-025572	7,131.76
Computers for Reflection High Energy Electron Diffraction	Sun Microsystems Micron Electronics	PO-025710	9,044.00
		PO-025713	2,673.00
An atomic force microscope	Digital Instruments	PM-103787	82,750.00
		Total	\$107,710.00

Appendix 2

Publications and Dissertations Completed Using These Facilities

Refereed Publications

1. J. Zhang, X. Deng, D. Swenson, S. A. Hackney and M. Krishnamurthy, "Formation of Nanoscale Trenches and Wires as a Pathway to Phase Separation in Strained Epitaxial Ge-Sn Alloys", *Thin Solid Films* 357 (1999) p. 85
2. X. Deng and M. Krishnamurthy, "Self-Assembly of Quantum-Dot Molecules Heterogeneous Nucleation of SiGe Islands on Si(100)", *Physical Review Letters* 81 (1998) p. 1473
3. X. Deng, J. D. Weil and M. Krishnamurthy, "Temperature Dependence of SiGe Coherent Island Formation on Si(100): Anomalous Re-entrant Behavior", *Physical Review Letters* 80 (1998) p. 4721
4. X. Deng, B.-K. Yang, S. A. Hackney, M. Krishnamurthy and D. R. M. Williams, "Formation of Self-Assembled Quantum Wires During Epitaxial Growth of Strained GeSn Alloys on Ge (100): Trench Excavation By Migrating Islands", *Physical Review Letters* 80 (1998) p. 1022
5. J. D. Weil, X. Deng and M. Krishnamurthy, "Preferential Nucleation of Ge Islands at Self-Organized Pits Formed During the Growth of Thin Si Buffer Layers on Si (110)", *Journal of Applied Physics*, 83 (1998) p. 212

Conference Proceedings

1. J. Zhang, D. Swenson, S. A. Hackney, "Fabrication of epitaxial pyramidal islands on Ge(100) Using Sn/C Lithographic Masks", Materials Research Society 1999 Fall Meeting, Boston, MA
2. J. Zhang, "Manipulation of Self-Assembled Nanostructures of Group IV Elements by Molecular Beam Epitaxy", Graduate Student Award presentation at the Materials Research Society 1999 Fall Meeting, Boston, MA
3. X. Deng and M. Krishnamurthy, "Role of Surface Instability and Anisotropy in Strain Relaxation of Epitaxial SiGe on Si(110)", Materials Research Society 1998 Spring Meeting, San Francisco, CA
4. J. Zhang, X. Deng, D. Swenson, S. A. Hackney and M. Krishnamurthy, "Formation of Nanoscale Trenches and Wires as a Pathway to Phase-

Separation in Strained Epitaxial Ge-Sn Alloys", Materials Research Society
1998 Fall Meeting, Boston, MA

5. X. Deng and M. Krishnamurthy, "Self-Assembled Quantum Dot Molecules",
Materials Research Society 1998 Fall Meeting, Boston, MA

Ph.D. Dissertations

1. Jie Zhang, "Self-Assembled Nanostructures of Group IV Elements on Ge
Substrates Using Molecular Beam Epitaxy", PhD Thesis, Michigan
Technological University, Spring 2001 (anticipated)
2. Xurui Deng, "Fabrication of Self-Assembled SiGe Quantum Nanostructures on
Si Surfaces Using Molecular Beam Epitaxy", PhD Thesis, Michigan
Technological University, 1999

Appendix 3

Publications Based on Application of the AFM to Measure Interfacial Forces

1. J. Drelich, J. Nalaskowski and J. D. Miller, "Preparation of Spherical Toner Particles for Atomic Force Microscopic Studies of Colloidal Forces," *Journal of Colloid and Interface Science* 201 (1998) 253-256.
2. J. Drelich, J. Nalaskowski, K.-Y. Chee, S. Veeramasuneni and J.D. Miller, "The Use of Atomic Force Microscope for Examination of Interfacial Forces in Wastepaper Deinking Systems," *Progress in Paper Recycling* 8 (1999) 55-67.
3. J. Pletka, A. Gosiewska, K.-Y. Chee, J.P. McGuire, J. Drelich and L. Groleau, "Interfacial Effects of a Polyalkylene Oxide/Fatty Acid Surfactant Blend in De-inking Flotation of Mixed Office Waste," *Progress in Paper Recycling* 9 (2000) 40-48.
4. J. Drelich, J. Nalaskowski, A. Gosiewska, E. Beach and J.D. Miller, "Long-Range Attractive Forces and Energy Barriers in De-Inking Flotation: AFM Studies of Interactions between Polyethylene and Toner," *Journal of Adhesion Science and Technology*, (2000) in press.
5. J. Drelich, J. Pletka, P. Boyd, E. Raber, D. Herron, E. Luhta, H. Walqui, N. Tervo, S. Boston, J. Wieland, J. Morgan and N. Sabo, "Interfacial Chemistry Aspects of De-Inking Flotation of Mixed Office Paper," Preprints of the 2001 SME Annual Meeting, SME 2001, Preprint # 01-9.
6. E. Beach, J. Drelich and R. Han, "Measurements of Adhesion Forces in Pharmaceutical Powder-Polymer Systems by Atomic Force Microscopy," in *Particles on Surfaces: Detection, Adhesion and Removal*, K.L. Mittal (Ed.), submitted (2000).
7. J. Drelich, E. Beach, A. Gosiewska and J.D. Miller, "Limitation of the Young-Dupre Equation in the Analysis of Adhesion Forces Involving Surfactant Solutions," *The Journal of Adhesion*, 2001, in press.
8. E. Beach, A. Gosiewska, Ch. Fang, K. Dudeck and J. Drelich, "Study of the Interfacial Effects of an Ethoxylated Alcohol Surfactant on the Flotation of Oil from Contaminated Soil," in *Environment Technology for Oil Pollution*, Vol. 1, J. Hupka and J.D. Miller (Eds), Technical University of Gdansk, Poland 1999, pp. 89-93.